IMPROVEMENT OF REINFORCED PLASTICS

January 19, 1962

Prepared under U. S. Navy, Bureau of Naval Weapons

Contract NOw 61-0613-d

Quarterly Progress Report No. 2

October 1 - December 30, 1961

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Prepared by

Space Sciences Laboratory
Missile and Space Vehicle Department
GENERAL ELECTRIC COMPANY
Philadelphia 4, Penna.

ABSTRACT

This report describes work performed during the second quarterly period (October 1 through December 31, 1961) on Contract NOw-61-0613-d, which calls for the development of hollow glass fiber reinforced composites and the evaluation of their mechanical and electrical properties.

Developments described in this report are (1) glass composition and hollow fiber manufacturing, (2) improved specimen fabrication techniques, (3) testing procedures and results.

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I. SUMMARY

Significant improvements in manufacturing of hollow glass fibers and specimen fabrication have produced extremely encouraging results. Tests on improved hollow glass fiber composites have shown compressive strengths as high as 110,000 psi for a strength to density ratio of over 2 million inches. Similar solid fiber specimens demonstrated strength to density ratios only as high as 1.3 million inches. This is a notable demonstration of increased structural efficiency through the reduction of material density.

Continued development of hollow fiber manufacturing processes has produced excellent configuration and uniformity of single fibers. Improved E-glass composition and impurity control have eliminated the boiling difficulties observed earlier, negating the need for conversion from glass to quartz.

II. GLASS MANUFACTURE

A. IMPROVEMENTS IN FIBERS

Because of the many difficulties encountered in the original plan of drawing 35 uniform fibers at one time, this plan was abandoned in favor of drawing a single fiber. It was felt that, once good results were obtained from this single fiber work, the number of fibers drawn could be progressively increased to the desired level.

This change in the program thus permits the production of improved fibers for test purposes and provides the opportunity for better understanding of the new techniques before drawing multiple hollow fibers. Prior attempts to draw multiple fibers resulted in unsatisfactory fiber configurations (see Figure 2). This variation ranged from solid cross-sections through extremely thin fiber walls.

Photomicrographs (Figure 1) are indicative of the uniformity of fiber dimensions produced by drawing one filament at a time. There is still room for improvement, however, in generating good fibers in other sizes and in collimation and packing of fibers. The glass fiber development work was done by the Lamp Glass Department of the General Electric Company in conjunction with the Missile and Space Vehicle Department.

The current filaments are being wound on 6" diameter Teflon bobbins with 1/4" x 1/4" cross-sections. Other winding techniques are being studied along with alternate schemes for shipping. Presently, deionized water is used to wet the glass fibers after drawing, and specimens are then shipped wet from Cleveland, Ohio, to Philadelphia, Pa. This water acts as a

lubricant and protects the delicate fibers during handling and shipping. After removal from the filament forming machine the glass-filled bobbins are sealed with a plastic film to retain this water until processed into samples in this laboratory.

Future plans are to produce hollow fibers with outside diameters of approximately 0.003" and 0.001". The inside diameter will be varied, with the thin fibers having at least 50% of the center area removed. Three outside diameters will be employed with constant ratio I.D./O.D., and for one diameter the I.D./O.D. ratio will be varied within 3 levels. Solid fibers will be made in each hollow fiber size for comparison purposes.

It is anticipated that modifications in the specimen fabrication procedure will be required for smaller diameter, less stiff fibers in order to maintain uniform collimation. The level of stiffness characteristic of the present fiber diameter (.0015") is adequate to obtain proper fiber collimation and orientation prior to molding.

B. GLASS COMPOSITION

E-glass manufactured by General Electric at Bridgeville, Pa. (designated type 172) has been used for the samples made during this reporting period. It has the following composition*:

SiO_2	60%
Al_2O_3	20%
CaO	7%
MgO	8%
NaO	1%
B_2O_3	4%

Its general properties are:

Specific gravity 2.53

Thermal expansion 42×10^{-7} cm/cm$^{\circ}$C, (0-300°C)

Softening point 914°C

*This is not a specification.

C. FIBER FINISH

Water is presently employed as a lubricant on the glass fibers. It is removed by drying at 250°F over night (15-18 hours) prior to resin impregnation.

It is thought that the water could deteriorate the glass fibers, dissolving some of the alkali from the surface of the glass. Also, after the water is evaporated from the glass, the resulting surface does not provide as good a coupling boundary between the glass and the resin as could be obtained using other treatments.

Various compounds such as Dow Corning 2-6030 silane monomers and DuPont Volan A will be investigated as sizing agents to provide a better coupling mechanism. Selection of these compounds was based upon their compatibility with the fiber forming process. Their use will not require difficult modifications to the present method of fabricating test samples. The need for these agents will then be established by measuring the bond weakening after immersion of specimens in boiling water.

III. SPECIMEN FABRICATION

A. MOLDING AND DRYING TECHNIQUES

In a previous report, mention was made of a technique for mechanically deforming the circular Teflon bobbin wound with hollow fibers (Ref. 3) into an oblong shape. This was tried with both hollow and solid fibers, and the results were very poor. Catenary action due to slack in the tightly wound fibers caused the supposedly straight sides to bow outward and destroy the collimation of fibers. It was impossible to maintain a uniform shape and fiber density throughout the "straight" portions of the oblong shape. Consequently a better technique which produces an excellent, straight specimen up to 15" long was devised.

This newly developed technique consists of immersion of the fiber-filled Teflon ring into water, then cutting radially through the fibers and ring at one point in the circumference. Stripping the wet hank of fibers from the ring is quite simple. Gently wiping the long hank between the fingers eliminates the catenaries formed due to initial relative winding diameter differences. This procedure thus achieves good collimation of the individual filament with little, if any, fiber damage. As indicated above, if smaller diameter fibers are employed this technique of collimation may or may not be applicable due to the possible lack of individual filament stiffness.

The hank of wet fibers is then inserted into the channel-shaped cross-section of an open end 15" long x 1/4" wide mold. Fiber alignment and integrity is maintained throughout the operation. A male plunger is positioned into the mold, exerting a nearly constant 0.6 psi pressure on the wet fiber bundle during an overnight drying process at 160°F. The dried glass is moderately bonded into a rectangular cross-section due to the intimate contact between the glass fibers. Higher molding pressures were obtained by placing weights on top of the plunger during the curing cycle.

The molds employed in this work were made from fairly soft aluminum. Because of the tight fit (.002" clearance) between the male and female sections, a few fibers could wedge between the mold surfaces and prevent proper pressure control. Use of different mold configurations and possibly the use of harder stainless steel molds is believed to eliminate this problem.

B. RESIN FORMULATION AND IMPREGNATION

The resin formulation employed in this project is as follows:

100 parts K opoxite 159 (resorcinal diglycidyl ether)

126 parts methyl nadic anhydride

25 parts EM207 (thiokol modifier)

1 part benzyldimethylamine

Minor variation in resin formulation may be necessary and desirable for compatibility with future applications of glass finishes. Excellent features of this resin system are low viscosity, high impact strength, high modulus and good transparency. Other advantages of the epoxy resin over a polyester system are less shrinkage and better adhesion to glass.

C. PREPARATION OF SPECIMENS

Preparation of the samples was accomplished by placing the pre-dried fibers and mold, at a temperature of 160°F, into a metal tray-like container. This in turn was placed on a hot plate at 160°F in a vacuum tank. The male plunger was shimmed up approximately 1/8" above the dried glass bundle, and resin at 160°F was poured into the container to a level above the dried fibers. Penetration of resin into the centers of the hollow fiber was prevented by maintaining the exposed fiber ends above the resin level. The tank was then sealed and evacuated for one hour.

After impregnation was completed, the mold was removed from the vacuum chamber and placed inside a curing oven. The curing cycle took place overnight at 200°F. Before curing, the mold shims were removed, and

weights were added to obtain the desired molding pressure. Removal of the part from the mold after curing was assured by the application of a mold release compound during the initial mold assembly.

D. PHYSICAL MEASUREMENTS

1. Specific Gravity

Specific gravity was determined from three one-inch long samples removed from the center and approximately one inch in from each end of a long specimen. Determinations were made on a weight and volume basis.

2. Resin Content

This was determined from before and after weight measurements, based on weight of resin loss, in a muffle furnace at 1000°F for three hours. There is no significant glass lost through this high heat cycle in the time allowed.

3. Photomicrographs

Samples approximately 1/8" long were removed from near the center of each molded specimen and imbedded in resin (this explains the apparent solid centers of the hollow fibers in Figure 1). After polishing, all samples were scanned optically. Photomicrographs of typical areas were taken at 300 X.

E. PROCESS VARIABLES

The major variables in this over-all process are listed below. Some are purposely controlled, others behave randomly.

1. Glass

a. Drawing rate and cooling rate
b. Consistency of glass (including chemistry)
c. Variation in O. D. and I. D.
d. Finish - type and degree
e. Handling and shipping factors
f. Continuity of fibers
g. Effect of moisture or solvents in handling of hanks
h. History of glass

2. Resin and Cure

a. Resin formulation
b. Shape of molded part

c. Temperature and duration of impregnation and cure cycles
d. Resin content of specimen
e. Pressure during cure
f. Mechanical properties of resin
g. Rate of cooling in restricted mold
h. Binding of mold surfaces

3. Sample Preparation and Measurements

a. Compacting and collimation of fibers
b. Location of samples in over-all specimen
c. Irregular end conditions (affecting test conditions)
d. Dimensions of specimen

IV. TESTING

The two tests used for rapid evaluation of glass fibers supplied during this fiber development portion of the program were (1) compression tests of short columns of hollow glass fiber reinforced composites and (2) tension tests of individual hollow fibers. It was felt that these two tests could screen the materials for basic strengths of weaknesses. Other, more specialized tests will be performed at a later date as superior hollow fiber materials become available in greater quantities.

A. COMPRESSION TESTS

Excellent strengths have been obtained on specimens manufactured from the most recent hollow glass fibers. Results from fibers made earlier in this reporting period show strengths considerably poorer than those now obtained.

Test specimens were cut from a long shaft of composite, formed as described earlier in this report. Two lengths were made, 3/4" and 3/8". Cross-head speed was .05"/min for the long ones and .025"/min for the short ones to provide nearly equal strain rates for the long and short specimens. Three specimens were removed from the shaft to test the density and resin content. One was taken from each end and one from the center of the shaft. The values obtained from these specimens were averaged to give a density which could be applied to the fractured test specimens to determine their individual structural efficiencies for compression, σ/ρ.

Specimens 1-H and 2-H (both hollow fiber reinforced) were molded using different molding pressures, thereby varying the resin content between the two specimens. Molding pressures were 0.66 psi for 1-H, and 4.55 psi for specimen 2-H and for later specimens 3-H and 1-S.

Specimens 1-H and 2-H represent early work on basic fiber screening and development. Specimens 3-H (hollow) and 1-S (solid) are representative of the best test specimens made during this reporting quarter.

The first part of Table I shows fairly poor performance of the two early specimens. Number 1-H had a low density and, considering its low stress at failure, displayed a maximum strength to density ratio of only 1.65 million inches. Notice that all of these specimens splintered vertically instead of exploding (type of failure observed in later tests to give highest strengths).

The next specimen, 2-H, although a relatively poor performer, did manage to explode in one instance. The highest strength to density ratio here was 1.2 million inches. As can be seen in the photomicrographs (Figure 1), the degree of hollowness is very low. The density, about 20% higher than the first specimen, confirms this observation.

The last half of Table I shows the test data from improved specimens resulting from the process development performed during this report quarter.

This next pair of specimens, numbers 3-H and 1-S, represent the latest effort in fiber development and specimen fabrication. Number 3-H is hollow and number 1-S is solid. Fiber sizes are nearly equal at .0015" O.D., as are the resin contents, but 3-H has 31% of the total specimen occupied by air. This means that nearly 45% of each glass fiber is removed by a large central hole whose diameter is about 67% of the outside fiber diameter.

Table I presents compressive test data which demonstrate the feasibility and potential for using hollow glass fiber reinforced composites as structural materials. In general, both hollow and solid fibers displayed nearly equal breaking strengths: hollow ones just slightly higher. But, the density of the hollow fiber composites is considerably lower than that for solid ones. This results in greatly increased strength to density ratios.

It is too early to speculate as to the eventual strength potential of the hollow fiber reinforced materials. Tests to be performed in the next quarter will evaluate other important mechanical, electrical, and thermal properties and also provide greater support for arriving at definite conclusions concerning the optimization of the hollow fiber geometries and specimen fabrication processes.

B. TEST OF INDIVIDUAL HOLLOW FIBERS

Several different glass fiber sizes were tested in tension to determine the effect of fiber diameter and wall thickness on specific strength of the glass.

Table II shows the breaking loads and the calculated stress at failure. Due to considerable scatter these data cannot be interpreted with absolute confidence; however, the trend is evident. Fibers with thinner walls and smaller diameters have higher strengths - all other things being equal.

Contrary to past beliefs, the ratios I. D. /O. D. and $t/\bar{D}$ seem to have no particular importance insofar as fiber tensile strength is concerned. Specific glass strength in the fibers tested varied roughly by a factor of 3.7 from control specimens A and C (Table II). The ratios I. D. /O. D. and $t/\bar{D}$ were held nearly constant throughout the tests. Hence the large strength variations cannot be attributed to these ratios. The most important parameters seem to be absolute diameter and absolute wall thickness.

It is felt that this data, although extremely valuable and interesting, is not sufficient to justify rash conclusions concerning the significant parameters that relate hollow fiber strengths to glass properties. Future work to be done in this area is intended to isolate and correlate any true relationships that exist among the fiber variables and their influence on fiber strength. This work is extremely important in order to establish optimum fiber parameters for maximum structural efficiencies.

V. PROBLEM AREAS AND PLANS FOR FUTURE WORK

Because of the excellent results obtained to date on the compressive strength measurements of hollow glass reinforced composites, it is felt that the program should now be directed toward systematically attacking the multitude of variables in the process. This requires a great quantity of specimens and tests of a number of material properties other than compressive strength.

1. Fiber production - Efforts will be made to produce a quantity of hollow fibers in 5 geometries: 3 with varying diameters and 2 with varying wall thicknesses. Solid fibers will be made in each of the 3 varying diameters to give easily compared data of hollow versus solid fibers. Special emphasis will be placed on uniformity of fibers and techniques. Fiber surface treatments will be studied in order to preserve the strength of the hollow fibers through impregnation and to provide good bonding with the resins. Studies will also be made of alternate schemes for shipping hollow fiber bundles before impregnation. Possibilities include (1) drying on spools before shipping, (2) cutting spools, laying straight, and drying prior to shipping on a stiff constraining rack and (3) use of other more inert liquids for immersion for protection of fibers during travel time.

2. Specimen fabrication - Difficulties outlined earlier in this report will be corrected through mold design studies. Attempts will be made to systematize the straightening of fibers now performed by hand wiping.

Smaller diameter fibers with less stiffness are expected to make this present process clumsy. Larger and more varied specimens will be molded for special testing requiring larger specimens.

3. Testing - Mechanical tests including longitudinal and transverse compression, hydrostatic compression, buckling, flexure and tension will be performed. Electrical and thermal properties will also be determined for selected specimens.

VI. REFERENCES

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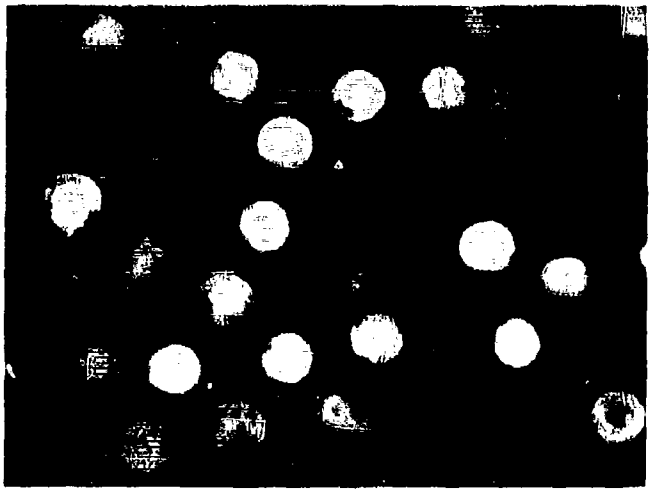
4. Pulos, J. G. and Buhl, J. A., Jr., "Hydrostatic Pressure Tests of an Unstiffened Cylindrical Shell of a Glass Fiber Reinforced Epoxy Resin," David Taylor Model Basin Report No. 1413, April, 1960.

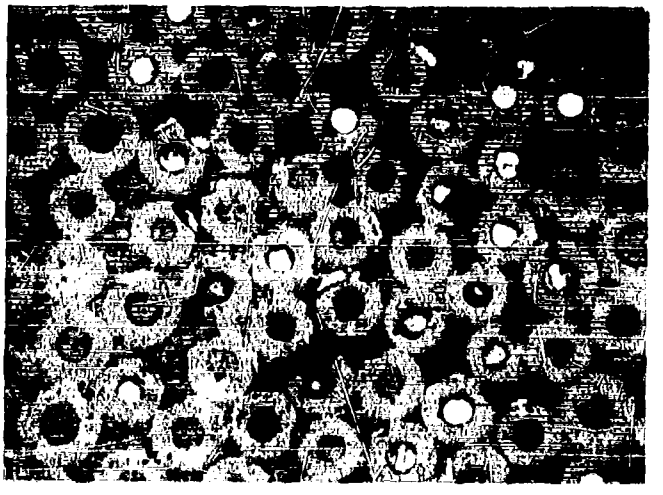


Weight Fraction
72.4% glass
27.6% resin

Volume Fraction
40.9% glass
31.1% resin
28.0% air

Specific Gravity 1.43

A. Hollow Fiber Specimen 1-H



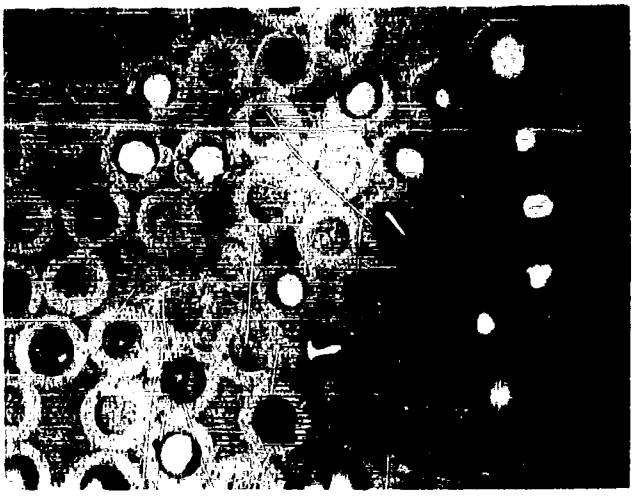
Weight Fraction
82.0% glass
18.0% resin

Volume Fraction
55.0% glass
20.3% resin
24.7% air

Specific Gravity 1.70

B. Hollow Fiber Specimen 2-H

Figure 1

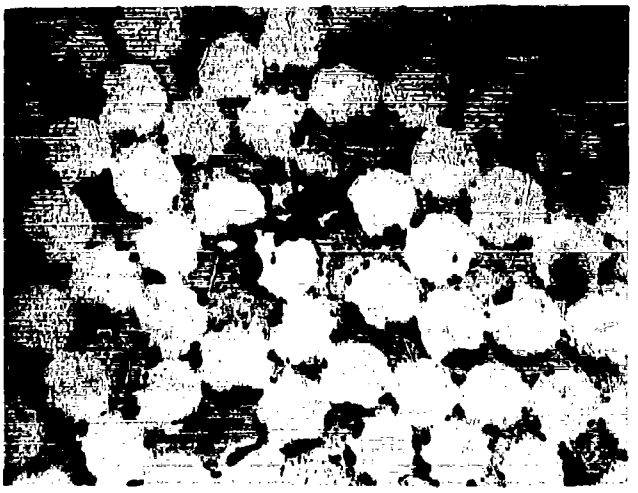


Weight Fraction
71.6% glass
28.4% resin

Volume Fraction
38.5% glass
30.4% resin
31.1% air

Specific Gravity 1.36

C. Hollow Fiber Specimen 3-H



Weight Fraction
80.3% glass
19.7% resin

Volume Fraction
67.9% glass
32.1% resin

Specific Gravity 2.06

D. Solid Fiber Specimen 1-S

Figure 1 (Continued)

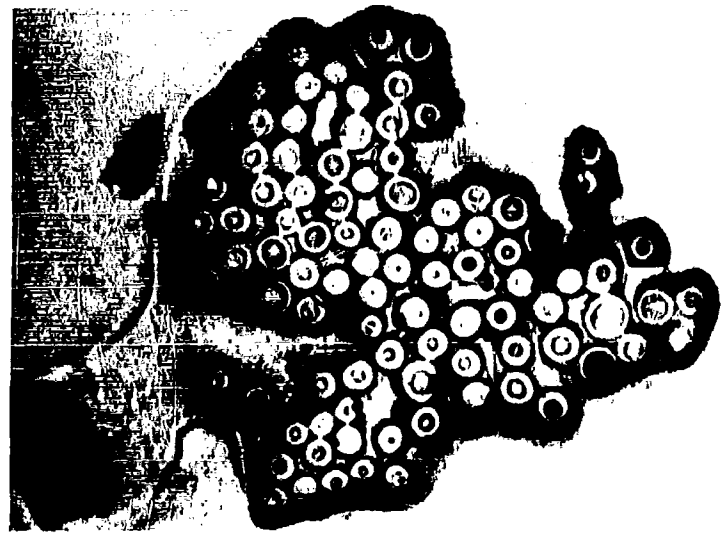


Figure 2. Example of Multiple Fiber Filaments Drawn Prior To This Report Period (Reference 3)

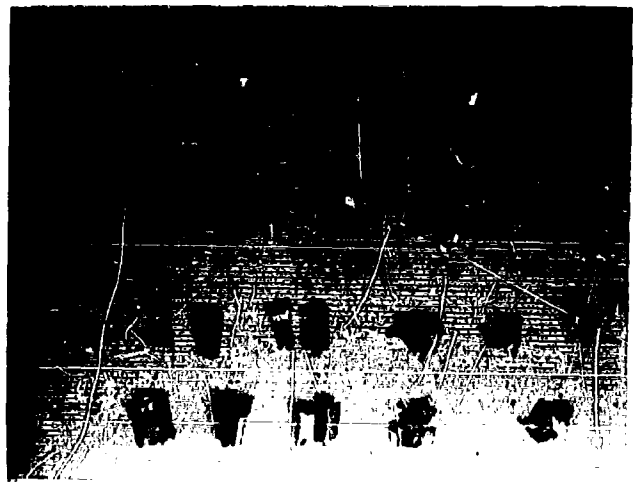


Figure 3. Broken Specimens After Compression Tests

TABLE I

PRELIMINARY COMPRESSION TESTS OF HOLLOW GLASS FIBER REINFORCED PLASTICS

Specimen Number	Width (in)	Thickness (in)	Length (in)	Area (sq. in)	Density (lb/in^3)	Load at Failure (lb)	Stress at Failure σ (psi)	Strength to Density Ratio (in)	Cross Head Speed (in/min)	Remarks on Failure Mode
Hollow Fibers										
1-H-1	.312	.259	.761	.0809	.0505	6700	82,900	1.64×10^6	.05	Mushroomed at top - splintered vertically.
1-H-2	.309	.261	.765	.0808	.0505	5740	71,000	1.405	.05	Mushroomed at top - splintered vertically.
1-H-3	.311	.260	.761	.0808	.0505	6240	77,200	1.529	.05	Mushroomed at top - splintered vertically.
1-H-4	.313	.258	.782	.0808	.0505	6730	83,300	1.650	.05	Mushroomed at top - splintered vertically.
1-H-5	.313	.257	.387	.0805	.0505	6450	79,800	1.580	.025	Mushroomed at top - splintered vertically.
1-H-6	.314	.257	.381	.0807	.0505	4310	53,200	1.053	.025	Mushroomed at bottom - splintered vertically.
	Average						69,500	1.476×10^6		
Hollow Fibers										
2-H-1	.258	.230	.760	.0594	.0604	1580	26,600	$.44 \times 10^6$	.05	Split vertically through center.
2-H-2	.260	.232	.754	.0604	.0604	2560	42,400	.704	.05	Mushroomed at top, then splintered vertically.
2-H-3	.259	.232	.761	.0601	.0604	2620	43,600	.722	.05	Mushroomed at top, then splintered vertically.
2-H-4	.262	.233	.378	.0611	.0604	4450	72,900*	1.205	.025	Exploded - split vertically and horizontally.
2-H-5	.261	.233	.378	.0609	.0604	2150	35,300	.585	.025	Bulged at center, then split vertically.
	Average						44,100	$.730 \times 10^6$		

Table I

(Continued)

Specimen Number	Width (in)	Thickness (in)	Length (in)	Area (sq. in)	Density (lb/in^3)	Load at Failure (lb)	Stress at Failure σ(psi)	Strength to Density Ratio (in)	Cross Head Speed (in/min)	Remarks on Failure Mode
Hollow Fibers (Control Specimen)										
3-H-1	.260	.176	.757	.0458	.0493	3705	80,900	1.64×10^6	.05	Mushroomed at bottom - split vertically.
3-H-2	.261	.175	.753	.0458	.0493	3575	77,900	1.58	.05	Mushroomed at top - split vertically.
3-H-3	.261	.176	.753	.0459	.0493	4880	106,500	2.16	.05	Mushroomed at top - split vertically.
3-H-4	.261	.175	.378	.0457	.0493	4500	98,300	2.00	.025	Mushroomed at top - split vertically.
3-H-5	.261	.175	.378	.0457	.0493	5050	110,500*	2.24	.025	Splintered edges and exploded.
3-H-6	.261	.175	.381	.0457	.0493	4630	101,000	2.05	.025	Mushroomed at bottom - splintered vertically.
	Average						95,800	1.94×10^6		
Solid Fibers (Control Specimen)										
1-S-1	.320	.257	.753	.0822	.0746	6600	80,400*	1.08×10^6	.05	Mushroomed at top, then exploded.
1-S-2	.320	.260	.752	.0832	.0746	6000	72,100	.97	.05	Mushroomed at top - split vertically.
1-S-3	.320	.259	.752	.0829	.0746	6230	75,400	1.01	.05	Mushroomed at top - split vertically.
1-S-4	.317	.260	.378	.0824	.0746	7810	94,900*	1.27	.025	Mushroomed at bottom - then exploded.
1-S-5	.318	.260	.382	.0826	.0746	6710	81,200	1.09	.025	Mushroomed at top - splintered vertically.
	Average						80,800	1.08×10^6		

*Ideal conditions result in exploding of test specimen.

TABLE II

TENSILE TEST OF INDIVIDUAL HOLLOW GLASS FIBERS

Specimen Size	O. D.	I. D.	t	I. D. /O. D.	$\bar{D}$	$t/\bar{D}$	Breaking Stress, S	Comments on Fibers
A	.00305"	.00224"	.000355"	.735	.002645"	.1585	20,000 psi	Virgin Fibers
							21,000 psi	
							50,000 psi	
					Average Strength		30,000 psi	
B	.00261"	.00192"	.000345"	.735	.002265"	.152	41,000 psi	Chilled with Water
							41,000 psi	
							50,000 psi	
							32,000 psi	
					Average Strength		41,000 psi	
C	.00135"*	.00097"	.00019"	.718	.00116"	.164	69,000 psi	Virgin Fibers
							145,000 psi	
							123,000 psi	
					Average Strength		112,000 psi	

*Approximate size used in compressive tests reported in Table I.

Nomenclature

O. D.	Outside diameter of fibers, inches
I. D.	Inside diameter of fibers, inches
t	Wall thickness of hollow fibers, inches
$\bar{D}$	Mean diameter (O. D. + I. D.) 1/2, inches
σ	Stress, based on gross area, psi
S	Stress, based on net glass area, psi
E	Modulus of elasticity, based on gross area, psi

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Mtls & Process Eng.

38. U.S. Rubber Co.
1700 E. Street, N.W.
Washington 6, D.C.
Attn: Mr. J.W. DeVorss

39. Natl Aeronautics & Space Adm.
Lewis Research Center
21000 Brookpark Road
Cleveland 35, Ohio
Attn: Library

40. Lockheed Aircraft Corp.
California Division
Sunnyvale, Calif.
Attn: E.A. Green,
Production Eng. Dept.

41. Commander
U.S. Naval Ordnance Test Station
China Lake, California
Attn: Mr. S.H. Herzog

42. Hdqtrs, Air Force Systems Command
Aeronautical Systems Division
Wright-Patterson Air Force Base,
Ohio Attn: ASRCEM-2
(Mr. T.J. Reinhart)

43. Electric Boat
Division of General Dynamics Corp.
Groton, Conn.
Attn: Robinson Research Library

44. Pittsburgh Plate Glass Co.
Glass Research Center
Box 11472
Pittsburgh 38, Pa.
Attn: R.M. Watterson, Librarian

45. Bureau of Naval Weapons
Special Projects Division
Attn: Mr. Harold Bernstein

46-49. Aerospace Industries Assn.
Shoreham Bldg.
Washington, D.C.
Attn: Mr. J.P. Reese

50. Aerojet-General Corp.
Azusa, California
Attn: Mr. Ed Rucks